NEW PATENTS

METALS AND ALLOYS

Palladium-Containing Austenitic Stainless Steel

MITSUBISHI JUKOGYO  European Appl. 458,606A

A Pd-containing austenitic stainless steel, for use in contact with concentrated H$_{2}$SO$_{4}$ at high temperature, comprises by wt. %: $< 0.004 - 0.04$ C, 5 - 7 Si, $< 2.0$ Mn, 15 - 25 Cr, 4 - 24 Ni, 0.01 - 1.07 Pd and the balance Fe. Unavoidable P and S impurities may be present in maximum amounts of 0.03 wt. % and 0.014 wt. %, respectively. The O content must be $< 50$ ppm, with the sum of O and S content not exceeding 150 ppm. The Pd-containing steel is used in absorption contact with concentrated H$_{2}$SO$_{4}$, at high temperature and cooling towers, pumps and vessels in the H$_{2}$SO$_{4}$ industry.

Ruthenium-Aluminium Intermetallic Compound

GENERAL ELECTRIC CO.  U.S. Patent 5,152,853

An intermetallic compound of Ru and Al comprises (in at. %): 40 - 51 Al, 0.8 - 9 Sc and B, balance Ru. It has a high temperature hardness at $\leq 1150^\circ$C and good room temperature toughness, comparable to that of Ti-24Al-11Nb alloy. It can be prepared by arc or induction melting, and by powder metallurgy methods. The compound can be used for high temperature applications, in jet engine components, and applications requiring a light weight and retention of good elevated temperature strength.

Palladium-Iron Electroresistive Alloy

DENKII JIKI ZAIRYO KENKYUSHO  Japanese Appl. 4/235,233

A Pd-Fe alloy comprises by atomic wt.%: 50 - 95 Pd, 5 - 50 Fe, 0.01 - 20 Mn, and small amounts of impurities, and has a temperature coefficient of resistance between room temperature and 200$^\circ$C of $\geq 3000 \times 10^{-6}^\circ$C. The alloy has a thermal output performance factor of $\sim 23$ times higher than that obtained with pure Pt.

CHEMICAL COMPOUNDS

Substituted Xanthates of Platinum, Rhodium or Iridium

EXXON RES. & ENG. CO.  U.S. Patent 5,068,375

Substituted platinum group metal xanthates contain alkyl, optionally substituted with alkoxy, or aryl, (un)branched alkylene, and Pt, Rh or Ir, with n = 2 when M = Pt, or n = 3 when M = Rh or Ir. Pt metal xanthates, such as bis-1-methoxy-2-propyloxanxhate, bis-3-methoxy-1-butyloxanxhate, etc., or Pt ethoxethylxanxhate or Pt butoxethylxanxhate were prepared. The compounds are used in catalyst systems for removing impurities, such as S and N, from hydrocarbons by hydrotreatment.

ELECTROCHEMISTRY

Electrolytic Manufacture of Ozone Using a Platinum Coated Anode

JOHNSON MATTHEY P.L.C.  European Appl. 470,761A

Ozone is prepared using an electrode made of W, Ti, Ta and Nb in contact with a second area which contacts the electrolyte comprising Pt or Pt containing material. The electrode is preferably W and/or Nb and the second area is Pt in a form to give at least one area of $\leq 30$ mm$^2$ which is either square, circular or rectangular. The electrolyte is preferably 0.01 - 8 N H$_{2}$SO$_{4}$ containing fluoride ions. The Pt catalytic coating prevents the formation of passive oxide layer.

Electroluminescent Electrode Containing Ruthenium Complex

CAPE COD RES.  U.S. Patent 5,075,172

An electrochemiluminescent (ECL) electrode for use in apparatus for monitoring the concentration of organic material in H$_2$O comprises an ECL layer of a homogeneous mixture of a tris(2,2'-bipyridyl)Ru complex and a PTFE polymer backbone with pendant side chains terminating with sulphonic groups, deposited on an etched glass substrate layered with a light transparent, electrically conducting, fine grained substance for supporting and electrochemically activating the ECL layer. The improved ECL layers are especially useful for monitoring petroleum contaminated ground H$_2$O.

Electrocatalytic Cathode for Chloralkali Electrolytic Cells

DOW CHEM. CO.  U.S. Patent 5,164,062

Cathode comprises a metallic surfaced substrate with a hard continuous non-dendritic coating of Pd, Ru, and optionally of Rh, Os, Ir and/or Pt, 1 - 3 $\mu$m in thickness, which is tightly adherent and thus is resistant to loss when the cathode is in use. The substrate is a mesh or woven screen of Ni, Fe, stainless steel, Cu and/or their alloys. The cathode is used to produce halogen gas, H$_2$ and alkali metal hydroxide solution by electrolysis of alkali metal halide solution.

Platinum Group Metals Coated Electrodes for Water Electrolysis

PERMELEC ELECTRODE LTD.  Japanese Appl. 3/253,590

The electrode is manufactured by applying a coating solution of a platinum group metal compound to one side of a porous electrode base maintained at a temperature near to the boiling point of the coating solution, heat-treating the coating solution at 80 - 200$^\circ$C to give a platinum group metal layer on the surface of the base, followed by a further PbO$_2$ catalyst layer to obtain an electrode. The electrode is used for ozone production and has the electrode substance only at the part that contributes to the electrolysis.
Platinum Containing Ionisation Catalyst Composition for Electrodes
TOKUYAMA SODA K.K.  Japanese Appl. 4/298,240
An ionisation catalyst composition for electrodes contains 50–99.9 wt.% Pt and 0.1–50 wt.% perovskite type oxide of La, Sr AO, where x = 0.01–0.5 and A is at least one of Co, Cu, Fe, Mn and Ni. In an example, the catalytic composition of 1 wt.% of perovskite type oxide was added to Pt black and made into a paste with terpineol, then screen printed to a thickness of 20 μm on both sides of the solid electrolyte. The catalyst is used for O ionisation electrodes in O₂ sensors and fuel cells.

ELECTRODEPOSITION AND SURFACE COATINGS

Platinum Electroforming or Plating Bath
ELECTROPLATING ENG.  European Appl. 465,073A
The plating bath comprises 2–100 g/l (calculated for Pt) of at least one of H₃(PTCl)₆, alkaline metal chloroplatinate, alkaline metal hexahydroxoplatinate, and 20–100 g/l of an alkaline metal hydroxide. The bath also contains soluble carboxylate, phosphate, sulphate and an alloying metal salt of Pd, Ir, Ru, Au and Ag. Electroplating or electroforming is carried out at ≥ 65°C with a current density of 1–3 A/dm², to form product of ≤ 50 μm thick. The Pt material has high hardness, thickness and improved corrosion and scratch resistance.

Ruthenium Coated Molybdenum Substrate Article
SKW METALS UK LTD.  European Appl. 508,721A
The article comprises a Mo-substrate with a Ru-coating which is directly coated onto the Mo-substrate. Ru is coated onto an intermediate layer of Ni, Ag, Au, Co, Cu, Cr, Rh or Pd, which is directly coated onto the Mo-substrate. The Ru coating is 0.15–0.8 μm thick. The Ru coated article has good resistance to oxidation and chemical attack and is used as a heat conductor in high power semiconductor devices. The Ru coating has high hardness, low electrical resistance and good abrasive wear resistance.

Adhesion and Porosity of Palladium Plating
AMERICAN TELEPHONE & TELEGRAPH CO.  European Appl. 512,724A
Plating an electrically conducting surface comprises electroplating at least two layers in succession, a Pd strike layer and a cover layer of a metal selected from Pd-Ni alloy, or Pt, Pd, Rh, Ru, Au, Ag and their alloys. The strike layer is deposited from an aqueous bath of pH 2–6 which comprises 0.1–3 g Pd/l and an organic diamine complexing agent. The acid Pd strike bath improves the adhesion and porosity of Pd (alloy) plating on metal substrates. The Pd strike protects corrosionable substrates from chemical attack in the main bath and prevents its contamination.

Precious Metal Composition
JOHNSON MATTHEY P.L.C.  European Appl. 514,073A
A homogeneous composition for forming, on firing, a film of noble metal (1) is selected from one or more of Pt, Pd, Au and Ag. When the composition is heated the H₂O evaporates to a homogeneous composition of polymeric resin, thiolate decomposes to (1) and the resin is volatilised. The firing is performed at 460–900°C leaving bright films. The composition is particularly suitable for application to ceramics.

Rhodium Plating for Copper Laminated Layered Plate
NIPPON AVIONICS K.K.  Japanese Appl. 3/223,486
Rhodium plating of a Cu laminated layered plate for slip rings is performed by subjecting a Cu foil surface to wet plating with Cu, Ni and Au before Rh plating. The Rh plating layer is 1.0–2.5 μm thick. The insulating layer comprises a glass cloth base and epoxy resin. Soft etching using NH₄ persulphate is effected between the Cu-, Ni- and Au-plating steps. Abrasion resistance is improved.

Electroless Palladium-Silver Alloy Plating Film Manufacture
EBARA CORP.  Japanese Appl. 3/287,781
Film is precipitated on a catalyst active carrier by using a plating bath containing at least one base of Pd salt, Ag salt, NH₄ and amine compound with Pd and Ag salts (0.001–0.5 mol/l), hydrazine and/or hydrazinium salt as reducing agent, amino-polycyclic acetic acid and/or its salt as chelating agent.

Probe Pin for Semiconductor Etching
TANAKA KIKINZOKU KOGYO K. K.  Japanese Appl. 4/236,439
A probe pin for semiconductor etching is prepared by dipping Ag-Pd alloy ultrafine wire tops in an electrolyte containing in g/l 10–100 of liquid KCl, 10–100 K ferrocyanide, 5–50 KOH and 0.5–5 ml/l surfactant, at a rate of 0.1–0.4 A/wire at 50–70°C. The wire was electrolysed to give a taper to the wire by etching as the diameter of the wire top is reduced gradually, by discharging the electrolyte or pulling up the wire gradually. The produced probe has high dimensional accuracy and stability.

Selective Plating of Non-Metal Substrate with Palladium Oxide Layer
CANON K. K.  Japanese Appl. 4/236,784
A non-metal substrate is plated selectively by forming a Pd oxide fine particle layer on the surface of the substrate, electron beam irradiating the Pd oxide at a desired location of the fine particle layer, for reducing to Pd metal, and dipping the substrate into an electroless plating bath for depositing a plating metal onto Pd as the nucleus.
Platinum Electroplating Bath for Sensors
TANAKA KIKINZOKU KOGYO K.K.
Japanese Appl. 4/297,592
A Pt electroplating bath consists of an aqueous solution containing Pt dinitroammine, citrate, sulphamic acid and NH₄ sulphamate. Glossy Pt coatings 15 μm thick can be obtained at a low (4–50°C) bath temperature. The plating bath is used to produce sensors and exothermic resistant materials with the characteristics of Pt, and electrodes of Fe, Cu, Al, Ni, Ti, Nb and Ta coated by Pt.

Electrolytic Deposition of Palladium-Nickel Alloy
KONTAKT SPEZIAL GDR.
East German Patent 292,596
The electrolytic deposition of a Pd-Ni alloy contact layer from an electroplating bath containing 10–15 g/l Pd and 5–10 g/l Ni salts, preferably in NH₄ solution, comprises adding a polyfunctional 5-membered heterocyclic compound to the bath. The invention allows the deposition of Pd-Ni alloy layers for production of printed circuit boards, decorative coating and gives constant alloy properties over a long period of operation.

APPARATUS AND TECHNIQUE
High Purity Thin Platinum Wire in Liquid Gas Sensor
OKAZAKI SEISAKUSHO
Japanese Appl. 3/276,053
A liquid gas sensor for rocket fuel comprises a resistor made of a high purity thin Pt wire contacted and wound on an insulating spool coated with a ceramic. By using a spool which is as small as possible, the heat capacity is reduced and the response increased. Ceramic coating on the Pt wire improves the mechanical strength. The sensor shows good response, high mechanical strength and can detect liquid H₂ used as rocket fuel. The response of the sensor is also raised by the use of the fine (thin) Pt wire of high purity.

FET Type Oxygen Sensor
SUZUKI MOTOR CORP.
Japanese Appl. 4/233,449
FET type O₂ sensor comprises solid LaF₃ electrolyte and Pt film catalyst for the gate metal electrode, and an OH⁻ ion implanted onto the surface. The reaction of O₂ to OH⁻ is very fast, as is the reaction of OH⁻ to LaF₃, thus improving the responsiveness of the sensor compared to the conventional products.

Sensor for Measuring Oxygen Partial Pressure in Molten Salt
NIPPON STEEL CORP.
Japanese Appl. 4/252,946
The sensor comprises a protection tube containing CaO and/or Y₂O₃, ZrO₂ and Pt powder, a Pt electrode dipped in the Pt powder, and a Pt electrode dipped in the molten salt outside of the protection tube. Both of the Pt electrodes are electrically connected, one inside and one outside of the tube.

Electromagnetic Flowmeter
FUJI ELECTRIC MFG. CO. LTD.
Japanese Appl. 4/254,715
Electromagnetic flowmeter consists of a ceramic tube for sample gas flow, and a pair of electrodes made of a Pt rod and a covering film. The Pt electrodes are coated with an oxide of Mg, Ca, Al, Si, Ti or Zr. The electrodes are produced by (1) application of an alcoholic solution of Mg, Ca, Al, Si, Ti or Zr to the outer surface of the Pt rod, and (2) heating the Pt rod to make the covering layer of the metal oxide.

Metal Oxide Semiconductor Oxygen Gas Sensor
TOYOTA JIDOSHA K.K.
Japanese Appl. 4/269,650
The sensor consists of an Al₂O₃ baseplate, a heater, a pair of Pt electrodes, a gas sensing layer of TiO₂, a reaction layer on the gas sensing layer, and a trap layer on the reaction layer. The trap layer traps impurities such as Pb, while the reaction layer is useful for the removal of the unburnt combustible gases.

Microporous Fluorine-Containing Resin Plated with Platinum
JAPAN GORE TEX. INC.
Japanese Appl. 4/286,637
The resin material has many pores of ≤100 μm, and Pt metal or Pt alloy film is bonded through anion exchange resin in the micropores. Preferably the Pt or platinum group metal alloy is plated by bonding a F⁻ containing resin containing sulphonic or carboxylic acids, immersing the textiles into an organic solvent, followed by boiling. Pt is deposited on the Pt nucleus by immersing the porous material in Pt containing solvent.

Oxygen Sensor for I.C. Engines
NGK SPARK PLUG CO. LTD.
Japanese Appl. 4/291,143
Oxygen sensor for I.C.E. is produced by forming a standard gas side electrode on the face of a solid electrolyte, and a detection gas side electrode on the other face. A thin film of Pt is formed on the surface of the solid electrolyte, with holes of 0.5–20 μm in diameter dispersed in it, and a layer of Rh, or an alloy of Rh and platinum group metal is coated on the Pt surface. The O₂ concentration is determined by using the e.m.f. formed from the difference in O₂ concentration between the standard and detection gas sides.

Air Intake Sensor for I.C.E.
NGK INSULATORS LTD.
German Appl. 4,207,188
Air intake sensor for I.C.E. has a Pt heat generating resistor and a Pt temperature compensating resistor in the same flow channel. The resistance value of the heat generating resistor must satisfy a specific function of the temperature, and the temperature coefficient of the effective resistance of the compensation resistor is less than that of the heat generating resistor. The sensor requires no temperature-compensating electrical circuit or circuit regulation and air flow can be accurately registered over a wide temperature range.
JOINING

Organic-Metal Conductor Composition for Ceramic Bonding
NORITAKE CO. LTD. Japanese Appl. 4/246,477

The conductor is composed of 1.0–10.0% Pt, 0.5–5.0% Pd, 5.0–25.0% Au, 0.02–1.0% Cr and 0.02–2.0% Bi, and an organic vehicle. It further contains organic Ag and V compounds with 0.5–5.0% Pd, 5.0–25.0% Au, 0.02–1.0% Cr and 0.02–2.0% Bi, and an organic vehicle. It is used for eutectic soldering for bonding.

HETEROGENEOUS CATALYSIS

Two Stage Upgrade of Aliphatic Hydrocarbons
BRITISH PETROLEUM P.L.C. British Appl. 2,246,141A

A feedstock comprising 2–8 C hydrocarbons is upgraded by contacting with dehydrogenation platinum group metal/silicalite catalyst at 550–650°C, and passing the product over a zeolite catalyst at 300–450°C to produce a second product rich in tertiary olefins and aromatics. The platinum group metal is Pt, Pd, Rh, Ir or Ru; a promoter such as Zn or Sn may be present. The zeolite catalyst is ZSM-5, promoted with Ga or Zn. Selectivity to aromatics can be controlled and production of (m)ethane is reduced.

Platinum Catalyst on Graphite Support of Specified Apparent Density
BASF A.G. European Appl. 467,174A

The catalyst contains 0.01–5 wt.% Pt on a graphite support with an apparent density of 1.7–2.2 g/ml. The graphite support has a compression strength of 50–250 N/mm², porosity < 20% and particle size of 1–600 µm. The catalyst is partly poisoned with S and/or Se. The catalyst is claimed for use as a hydrogenation catalyst, especially for the production of hydroxylation monosulphides by catalytic hydrogenation of NO in aqueous mineral acids. It has a long lifetime and high selectivity, is very active and gives high yields.

High Temperature Steam Reforming of Hydrocarbons
NIPPON OIL K.K. European Appl. 503,653A

The catalyst comprises 0.1–2 wt.% Ru supported on a carrier made of 5–40 wt.% CeO₂ and rare earth oxide, and 60–95 wt.% Al₂O₃, with the atomic ratio of Ce:Ru being 10–200. The catalyst has a preferred surface area of 5–200 m²/g and a porosity of 0.05–1.0 cm³/g. The steam reforming reaction is performed preferably at 500–600°C at the catalyst bed inlet and 700–800°C at the catalyst bed outlet with a steam:C ratio of < 2.5. The catalyst exhibits high activity, long catalytic life and low C precipitation even when used at comparatively low pressure and steam:C ratio.

Platinum Group Metal Catalyst for Hydrogen Peroxide Production
MITSUBISHI GAS CHEM. CO. INC. European Appl. 504,741A

H₂O₂ is prepared by direct reaction of O₂ and H₂ in a reaction medium containing a promoter in the presence of a platinum group metal catalyst, preferably Pt or Pd, supported on solid acid (SA) or solid superacid carriers (SSAC). SSAC is for example H₂SO₄/Al₂O₃, TiO₂ or ZrO, etc., and SA is a proton form of mordenite or of MFI type zeolite. The reaction medium is preferably an aqueous solution containing a promoter and a H₂O₂ stabiliser of aminotri(methylene phosphonic acid), etc. The reaction occurs in the presence or absence of a non-reactive gas at 50–50°C and 3–150 mg/cm² G pressure.

Three-Way Palladium Supported Catalyst for I.C.E.
NIPPON SHOKUBAI CO. LTD. European Appl. 507,590A

A catalyst for simultaneous removal of CO, HC and NOx from exhaust gas of I.C.E. is supported on a monolithic carrier and comprises 0.5–30 g Pd/Al catalyst, alkaline earth metal oxide, CeO₂, ZrO and activated Al₂O₃. The exhaust gas purifying system has on the exhaust gas inlet side the Pd catalyst, and on the exhaust gas outlet side a catalyst of Rh and Pt, or Pt, Pd and Rh on a monolithic carrier and a refractory inorganic oxide.

Platinum Metals Rev., 1993, 37, (3) 179
**Palladium Catalyst for Production of Difluoromethane**

**IMPERIAL CHEM. IND. P.L.C.**

*European Appl. 508,660A*

Difluoromethane is manufactured by the reaction of chlorodifluoromethane with H₂, by feeding a stream through a vessel containing a hydrogenation catalyst comprising 8-15 wt.% Pd and at least one other Group VIII metal, preferably Ni, on an active C support. The Pd loading on C is such that at 250°C, atmospheric pressure and a Pd loading of 10% w/w, the yield is ≥ 50%.

**Palladium/Carbon Catalyst for Preparation of Diaminobenzotrifluoride**

**OCCIDENTAL CHEM. CORP.**

*European Appl. 509,446A*

3,5-Diaminobenzotrifluoride was prepared by reacting 4-chloro-3,5-dinitrobenzotrifluoride, in MeOH, with H₂ in the presence of MgO and 5 wt.% Pd/C catalyst, used at levels of 0.5, 1, 4 or 6 g (dry weight) per 100 g, at ≤ 125°C. The fluoride is an intermediate in the synthesis of polyimide polymers and the process produces less that 0.5% of 4-chloro-3,5-diaminobenzotrifluoride.

**Lower Polyols Production with Platinum-Based Carbon Supported Catalysts**

**MENON SRL**

*European Appl. 510,238A*

The highly selective process comprises (a) contacting a mixture of carbohydrates and H₂, under pressure and weakly basic conditions, preferable with 1-7 wt.% Pt/C catalyst and a basic promoter, to give higher polyols, (b) hydrogenation of the higher polyols by contact with a Pt/C catalyst modified with sulphides to produce a mixture of lower polyols.

**Platinum-Iridium-Tin Catalyst for Low Pressure Semi-Regenerative Reforming**

**EXXON RES. & ENG. CO.**

*European Appl. 512,766A*

A process for catalytically reforming hydrocarbonaceous feedstream, such as naphtha and straight run gasoline, in the presence of H₂ at 1.75 x 10⁵-1.4 x 10⁷ kg/m³ and gas rate 43-142 m³/barrel comprises contacting the feedstream with a catalyst containing 0.1-1.0 wt.% Pt, 0.1-1.0 wt.% Ir and 0.02-0.4 wt.% Sn, preferably supported on Al₂O₃. The process improves the octane quality, is run at relatively low operating pressures and low gas recycle rates.

**Halogenated Hydrocarbon Fluorination Process with Ruthenium or Platinum Containing Catalysts**

**DAIKIN IND. LTD.**

*European Appl. 516,000A*

A halogenated hydrocarbon such as 1,1,1-trifluorochloroethane, etc., is fluorinated by reaction with HF in the presence of a catalyst comprising Cr oxide supported on a carrier, such as fluorinated Al oxide or Al fluoride, containing 0.01-10 mol% of Pt or Ru based on Cr oxide, at 290-380°C.

**Preparation of Noble Metal-Alloyed Catalysts**

**INT. FUEL CELLS**

*World Appl. 91/195,566A*

A noble metal alloyed catalyst with high loading comprises precipitating Pt and Pd compounds from the solution onto a support, reducing the catalytic activity by reducing the pH or temperature of the solution, adding a soluble alloying metal compound to the solution, precipitating it onto the support, and then calcining to form the alloyed catalyst. High and predictable loadings with reduced waste are obtained and are used in the preparation of fuel cell catalysts.

**Platinum Catalysts for Amino-Phenol Production**

**E. I. DU PONT DE NEMOURS & CO.**

*World Appl. 92/18,445*

3-Aminophenol was produced by dehydrogenation of 3-amino-2-cyclohexene-1-one in the presence of Pt or Pd-Pt catalyst supported on C, SiO₂, or an alkaline support, in a solvent with a catalytic amount of base in the reaction mixture, followed by heating at 130-350°C. The phenol produced by this method, after removal of the catalyst, can be used directly for the synthesis of 3-amino-diphenyl ethers.

**Platinum or Palladium Catalyst for Carbon Monoxide Oxidation**

**PHILLIPS PETROLEUM CO.**

*U.S. Patent 5,071,818*

The catalyst comprises a SnO₂ coated porous ceramic support, Pt and/or Pd, and Mn and/or Cr compound co-promoters. The support is obtained by impregnation with Sn carboxylate solution, then drying and heating to convert the carboxylate to SnO₂. The ceramic material is preferably a monolith.

**Palladium Catalyst in Diaminobenzotrifluoride Production**

**OCCIDENTAL CHEM. CORP.**

*U.S. Patent 5,144,076*

3,5-Diaminobenzotrifluoride is prepared in a single step by reduction of 4-chloro-3,5-dinitrobenzotrifluoride with H₂ in solvent, such as 3-6C secondary and ternary alcohols, acetonitrile, etc., in the presence of a Pd catalyst supported on an inert carrier and a base, such as alkali metal and alkaline earth metal carbonate and bicarbonate, and alkali metal salts of 2-6C aliphatic carboxylic acids. The trifluoride produced is an intermediate in the synthesis of polyimide polymers.

**Hydrocarbon Feedstock Hydrogenation Using Mordenite Supported Catalyst**

**AMOCO CORP.**

*U.S. Patent 5,151,172*

Hydrocarbon feedstock consisting of material boiling at 150-700°F is hydrogenated with H₂ in the presence of a catalyst comprising 0.1-2.0 wt.% each of Pd and Pt on a mordenite support. The mordenite has Si/Al atomic ratios of 10-40 and Pd/Pt wt. ratio is 5:1-1:2. The method gives improved deaomatisation, desulphurisation and denitrogenation of distillate feedstock.
Removal of Carbon Monoxide and Oxygen from Hydrocarbon Feeds
PHILLIPS PETROLEUM CO. U.S. Patent 5,157,204
Removal of CO and free O, from HC-containing feeds comprises contacting at -30 to 200°C, a fluid feed containing 0.1 mole ppm-6 mole %CO, 0.05 mole ppm-3 mole O, and at least one 1-10C HC with at least one Pt containing catalyst composition. The catalyst is selected from a composition containing: (a) Pt metal, Fe oxide and at least one inorganic support, such as Al₂O₃, MgO, etc., or (b) Pt and Pd, at least one Cr or Mn compound, and a SnO₂-coated porous ceramic monolith support. The catalyst is used for purification of ethylene and propylene for use as feeds for catalytic polymerisation processes.

Diesel Engine Exhaust Deodorising Catalyst
TOYOTA JIDOSHA K.K. Japanese Appl. 3/262,541
A platinum group metal catalyst for deodorising diesel engine exhaust consists of a refractory support having a zeolite surface layer loaded with Pt, Pd, Rh, Ru or Ir. In an example, a cordierite monolithic support was immersed in an aqueous slurry containing H₂PtCl₆ solution, and then in aqueous Pt(NH₃)₄OH solution. The catalyst comprises depositing exhaust-purifying catalysts consisting of Pt and Sr supported on y-Al₂O₃ and Sr₂O₃ supported catalysts giving high thermal resistance.

Chemical Heat Pump System
Y. SAITO Japanese Appl. 3/263,550
A 2-propanol/acetone-H system for a chemical heat pump comprises 0.1-20 wt.% supported platinum group metal, selected from Pt, Rh and Ru on activated carbon as a dehydrogenation catalyst for 2-propanol. 2-Propanol is quickly and effectively dehydrogenated in this pump, permitting the use of low temperature heat sources at < 80°C. The catalyst also allows effective recovery and re-use of heat energy from solar energy, geothermal heat and industrial waste heat.

Platinum Group Metal Catalyst for Purification of Exhaust Gas
CATALER KOGYO K. K. Japanese Appl. 4/256,445
Purification catalyst for exhaust gas comprises a zeolite impregnated with Co at inlet side of exhaust gas and zeolite impregnated with Pt, Pd, Rh, Ir or Ru at outlet side of exhaust gas. In an example, a cordierite honeycomb support is immersed in a zeolite slurry, in Co acetate solution, and in tetra Pt ammine solution to form the catalyst. It shows a 50% NO purifica- tion ratio at air/fuel ratio of 17, at 200°C.

Palladium-Iodine Catalyst for Continuous Production of Diaryl Carbonates
ASAHI CHEM. IND. CO. LTD. Japanese Appl. 4/261,142
Diaryl carbonates are prepared by the reaction of aromatic hydroxyl compounds, preferably phenols, with CO and O₂ in the presence of a Pd compound and promoter, I compound, catalyst at 50-350°C under 1-350 kg/cm². The reaction gives high yield and selectivity from available materials without toxic phosgene production.

Triple Function Exhaust Gas Purification Catalyst
CATALER KOGYO K. K. Japanese Appl. 4/265,155
The catalyst comprises a metal plate honeycomb coated with more than two layers of Al₂O₃, at least one of Pt, Pd, Rh and Ce with the first Al₂O₃ layer, which directly contacts the metal honeycomb plate, not containing Ce. This triple functional catalyst is used for removal of hydrocarbons, CO and NOx from the exhaust gas of internal combustion engines; the expansion of honeycomb metal base plates is prevented. The purifying capacity of this catalyst for hydrocarbons, CO, and NOx was 96-99%, 69-74% and 72-77%, respectively.

Palladium-Cobalt-Molybdenum Hydrogenation Catalyst
TONEN CORP. Japanese Appl. 4/265,157
Pd-Co-Mo/SiO₂-Al₂O₃ catalyst is produced by immersing the carrier in aqueous solution containing an amine complex of Pd and Co, and then in aqueous Mo compound solution. In an example, the catalyst contains 10 wt.% SiO₂ and carried 1.0 wt.% PdO, 35 wt.% CoO and 14 wt.% MoO₃.

Ruthenium Catalysts for Preparation of Heavy Aliphatic Hydrocarbons
AGENCY OF IND. SCI. & TECHNOL. Japanese Appl. 4/271,837
The catalyst contains 0.1-10 wt.% Ru, V in amount 0.1-20 times the atomic ratio of Ru, and TiO₂, and is used for synthesising heavy aliphatic hydrocarbons with > 12C from H₂ and CO. The heavy aliphatic hydrocarbons are produced at a high production rate and with high selectivity. The transformation ratio of CO to hydrocarbons was 9.1 x 10⁴ mol/(min g of the catalyst) yielding hydrocarbons: 31.6 wt.% C₁-C₄, 43.4 wt.% C₅-C₁₀ and 25.0 wt.% > C₁₂.

I.C.E. Exhaust Purification Using Platinum and Strontium Supported Catalyst
TOYOTA JIDOSHA K.K. Japanese Appl. 4/284,824
The method for purifying I.C.E. exhaust gas comprises depositing exhaust-purifying catalysts consisting of Pt and Sr supported on γ-Al₂O₃ or La-added Al₂O₃, in exhaust gas containing an excess of O₂ to purify NOx in the exhaust. The catalyst contains 0.5-2 wt.% Pt and 5-20 wt.% Sr, of the carrier and carried Pt, respectively.

Highly Thermal Resistant Catalyst for I.C.E. Purification
NE CHEMCAT K.K. Japanese Appl. 4/284,847
A catalyst for exhaust gas purification for I.C.E.s consists of a unitary structured carrier containing at least one platinum group metal, such as Pt or Rh; Al₂O₃, CeO₂, and stabilised ZrO₂, which contains Group IIa and IIIb elements, Y, La, Pr and Nd. The oxides of the alkaline earth metals and Zr are used in triple functional catalyst giving high thermal resistance.
Hydrogen Peroxide Production Using Platinum Catalyst

MITUSBISHI GAS CHEM. CO. INC.

*Japanese Appl. 4/285,003*

H₂O₂ was produced in a reaction medium using a Pt catalyst loaded on La oxide support in an aqueous acidic solution containing phosphoric acid and chloride ions; the chloride ions are used as an auxiliary catalyst. The process gives a higher concentration of H₂O₂ solution without dissolving the La oxide support.

Aminomethyl-Trialkylcyclohexylamine Preparation

MITUSBISHI KASEI CORP. *Japanese Appl. 4/300,852*

3-Aminomethyl-3,5,5-trialkylcyclohexylamines were prepared by catalytic hydrogenation of 3-cyano-3,5,5-trialkylcyclohexylamine in the presence of supported Ru catalyst and NH₃ at 50–150°C and 20–150 atm. The catalyst contains 0.1–10 wt. % Ru and is supported on SiO₂, Al₂O₃, or active C. The compound is useful as a curing agent for epoxy resins or urethane elastomer; the Ru catalyst is safer and more effective than former catalysts.

Titanium Dioxide/Palladium Catalyst for Hardening Fatty Acids

DEGUSSA A.G. *German Appl. 4/109,502*

A high activity TiO₂/Pd catalyst for hardening fatty acids consists finely divided Pd and pyrogenic TiO₂ of rutile and anatase structure obtained by flame hydrolysis of TiCl₄. The catalyst is prepared by forming the pyrogenic TiO₂ with conventional additives into a green body, drying at 110°C for 12 h, and impregnating the calcinated body with an aqueous Pd salt solution adjusted to pH 4 to obtain the final catalyst containing 0.5–10 wt. % Pd.

Palladium-Ruthenium Membrane Catalyst

PETROCHEM. SYNTHESIS INST.

*Russian Patent 1,694,638*

Technical grade fats for soap production are prepared by catalytic hydrogenation of vegetable oil, at 50–200°C, in a liquid phase, using Pd-Ru alloy membrane catalyst with a porous surface layer with 0.000125–0.0125 mg/cm² active particles. The latter consists of a melt of Pd and Ni, with 5–20 wt. % Ni. The membrane catalyst is a spiral pipe.

HOMOGENEOUS CATALYSIS

Preparation of Thermally Labile Vinyl Compounds Using Ruthenium Compound Catalyst

UNION CARBIDE CHEM. & PLASTICS

*European Appl. 506,070A*

Thermally labile Bronsted acid vinyl derivatives are prepared by transvinylolation. This comprises reacting a vinyl derivative of a first Bronsted acid with a thermally labile Bronsted acid in the presence of a transvinylolation Ru compound catalyst.

Preparation of Acetic Anhydride in the Presence of Rhodium Catalysts

SOLLAC S.A. *European Appl. 511,038A*

Acetic anhydride is prepared by the reaction of methyl formate with a gas comprising principally CO under a pressure of ≥ 1 MPa in the presence of (a) a Rh-based catalyst, (b) two I-based promoters of successively ionic and covalent characteristics and (c) an N-substituted cyclic amide as the solvent. Examples of Rh-based catalysts are RhCl₃, Rh₂O₂Cl₂, etc. The nature of the solvent is critical, since in a conventional solvent (or absence of solvent) the selectivity of acetic anhydride is decreased due to isomerisation of methyl formate to acetic acid.

Iridium-Based Catalyst for Adipic Acid Preparation

RHONE POULENC CHIM. *European Appl. 511,126A*

Adipic acid was prepared by the reaction of H₂O and CO with at least one pentenic acid in the presence of an Ir-based catalyst and at least one I promoter at a high temperature and at greater than atmospheric pressure. The Ir catalyst is preferably (IrCl₃(cod))₃, Ir(CO)₅, or Ir(acac)(CO)₃, where cod = cyclooctadiene, acac = acetylacetone, and the I promoter is H₂ or an organic iodide capable of generating HI. The atomic ratio I/I₄ is < 10. The reaction temperature is 100–240°C and the partial pressure of CO is 2–250 bar.

Hydrogen Peroxide Synthesis over Palladium Catalyst

INTEROX INT. S.A. *World Appl. 92/15,520A*

H₂O₂ is obtained by direct reaction of H₂ and O₂ in the gas phase at > atmospheric pressure over a Pd heterogeneous granular catalyst which is suspended in a liquid aqueous phase, and comprises a Pd metal compound or a combination of Pd with at least one other noble metal, impregnated onto an alkaline earth sulphate or phosphate support. The catalyst preferably contains 0.1–15 g per 100 g total metal and support. The process gives high selectivity of conversion of H₂ into H₂O₂ and overcomes any tendency of the catalyst support to dissolve in the medium.

Continuous Adipic Acid Manufacture with Halide Promoted Rhodium Catalyst

E.I. DU PONT DE NEMOURS & CO. *World Appl. 92/16,476A*

Adipic acid is prepared by contacting 1,3-butadiene, allylic butenols or allylic butenol esters or ethers with CO, H₂O and with a Rh catalyst and a halide promoter at 50–250°C and CO partial pressure of 100–3,000 psi to form a reaction mixture containing adipic acid and branched 6C dicarboxylic acids. Adipic acid is recovered from the mixture, and the remainder is heated to isomerise the dicarboxylic acids to adipic acid, and the production mixture is recycled. The molar ratio of promoter to Rh = 1–20:1.
Acetic Acid Production Using a Rhodium Catalyst

HOECHST CELANEOE CORP. U.S. Patent 5,144,068
Acetic acid is produced by the reaction of MeOH with CO in a liquid medium containing a Rh catalyst and 2-20 wt.% of a soluble iodide salt catalyst stabiliser of low H₂O content, giving >99% yield. The reaction medium contains 200-1000 ppm of Rh catalyst, preferably 1-10 wt.% H₂O, 0.5-5 wt.% methyl acetate, 10-20 wt.% LiI and 12-16 wt.% MeI. The process converts alcohols into the corresponding carboxylic acids, and gives a great degree of stability.

Unsymmetric Urea or Carbamate Preparation with Ruthenium Catalyst

ARCO CHEM. TECHNOLOGY LP U.S. Patent 5,155,267
Unsymmetric urea or carbamates are produced by contacting a primary (aromatic) formamide and a dialkylamine or alcohol with Ru, Pt or Rh compounds, especially RuCl₃(PPPh₃) or RuCl₂(DMSO)₃, in an aromatic hydrocarbon solvent at < 150°C. The products can be cracked to give isocyanates. The process is selective giving > 90% of the desired product.

Direct Oxidation of Benzene for Phenol Preparation

IDEMITSU PETROCHEM. K.K. Japanese Appl. 3/261,735
Phenols are prepared by reacting benzene with O₂ in the presence of Pd catalyst, such as Pd acetate, Pd black, Pd/C, Pd hydroxide, or colloidal Pd, under the conditions that the ratio of the partial pressure of O₂ to that of the catalyst to benzene is 1,500-20,000 kg/cm²/mol %.

Aminopropyl Alkoxy silane Preparation

SHOWA DENKO K.K. Japanese Appl. 4/210,693
Aminopropyl alkoxy silanes are prepared by reacting hydroalkoxy silane containing ≤ 10 mol. Cl equivalent to Rh in the catalyst, with allyl amine compound in the presence of a catalyst of a Rh compound and a trivalent P compound. The Rh compound is preferably RhCl₃, 3H₂O, Rh(acac)₃ and P compounds are P(OPh)₃, P(Et)₃, etc. Aminopropyl alkoxy silane can be obtained with high yield and high selectivity.

FUEL CELLS

Gas Reaction Fuel Cell with a Platinum Catalyst

U.S. DEPT. ENERGY World Appl. 92/15,121A
A H₂-O₂ fuel cell has a solid polymer electrolyte, separate anode and cathode, with a composite film of a supported Pt catalyst uniformly dispersed in a proton conducting ionomer disposed between the solid polymer electrolyte and the cathode. The Pt loading in the composite film is < 0.35 mgPt/cm² and the film is < 10 µm. The composite film optimises proton access to the catalyst while giving adequate gas permeability and electronic continuity.

CORROSION PROTECTION

Stainless Steel Alloy Containing Platinum Group Metals

GENERAL ELECTRIC CO. U.S. Patent 5,147,602
A corrosion resistant stainless steel alloy contains in wt.% 24-32 Cr, 20-40 Ni, ≤ 10 Mn, ≤ 0.06 C, 2-9 of at least one of Ti, Nb and Ta, a platinum group metal and balance Fe. The amount of platinum group metal, preferably 0.01-5 at.% of the alloy, is sufficient to reduce the corrosion potential of the alloy in high temperature H₂O containing H. The alloy has a high resistance to corrosion and stress cracking in high temperature H₂O as is found in H₂O deaerators, nuclear reactors and steam driven power stations.

Electrochemical Sensor for Corrosion Monitoring

HITACHI K.K. Japanese Appl. 3/218,452
An electrochemical sensor for monitoring corrosion of Zr alloy is made by using a Zr alloy, acting as the sample electrode, a reference electrode and Pt sensor electrode which is fixed in a ceramic mould and sealed. The whole is unified in a corrosion resistant stainless steel casing by diffusion bonding. The corrosion of Zr alloy in service is surmised from the sample alloy and the measurement of corrosion rate permits the deterioration in quality of the environmental H₂O to be determined.

Corrosion-Resistant Insoluble Electrode Containing Iridium Dioxide

NIPPON STEEL CORP. Japanese Appl. 4/214,899
A corrosion-resistant insoluble electrode comprises a valve metal layer formed on the electrode body, with IrO₂ reactive sputter coating formed in the valve metal layer, an IrO₂ baking film layer is formed on the sputter coating, and IrO₂ reactive sputter coating is further formed on the baking film layer. The application/drying cycle is performed 8 times, to give an IrO₂ layer 5 µm thick. The electrode was used as an anode in 5 wt.% H₂SO₄ at 60°C to test the corrosion using Pt as cathode. The insoluble electrode gives good corrosion resistance and durability under the high current density of electrolysis of ≤ 200 A/cm².

GLASS TECHNOLOGY

Process for Rhodium-Platinum Bushing Production

TOKURIKI HONTEN K. K. Japanese Appl. 4/241,105-6
A Rh-Pt bushing process for fibre formation includes a welding nozzle (10% Rh-Pt alloy wire) element to the centre of bushing bottom plate firmly, with Pt pipe fitted to the foot of the nozzle. Process makes a Pt plate layer on the surface of the bottom plate consisting of the following: Pt-Rh/Pt-Ru, Pt-Rh/Pt-Ir or Pt-Rh/Pt-W. The nozzle is welded to the bottom plate by heating, can be positioned at the centre of the hole accurately and fixed firmly.
Superlattice for Magnetic Recording Medium
OKI ELECTRIC IND. K.K.
European Appl. 459,411A and 459,413A
A magnetic recording medium comprises a substrate and an artificial superlattice of alternate layers of Co and Pt or Pd, comprising a vertical magnetisation film. Preferably, each Co layer is 1-15 Å thick and each Pt or Pd layer is 2-30 Å thick. The thin film has thickness 10-100 Å inclusive. The medium displays high residual flux density, high coercivity and low Curie temperature.

Magnetic Recording Medium with Cobalt-Palladium-Chromium Alloy
FUJI PHOTO FILM CO. LTD. European Appl. 505,783A
A magnetic recording medium comprises a non-magnetic substrate and a magnetic thin film of (Co90-Pd10)100-Cr, or (Co90-Pd10-Cr50)100-O, where x = 10-40, y = 5-25 and z = 0.5-15. A non-magnetic layer of thickness 200-10,000 Å is preferably included between the magnetic thin film and substrate. The magnetic thin film has thickness 200-10,000 Å and the film has perpendicular easy axis of magnetisation.

Manufacture of Opto-Magnetic Recording Films
HITACHI LTD. Japanese Appl. 4/186,706
Opto-magnetic recording films are manufactured by laminating a layer containing at least one of Pt, Pd, Rh and Au and a layer containing Fe, Ni and Co. The layers are laminated in turn to make a multilayered structure and the internal stress is controlled in order to control the anisotropic perpendicular magnetic energy. The film allows more stable storage of written information.

Sliding Contact Material
TOKURIKI HONTEN K.K. Japanese Appl. 4/235,291
The sliding contact material is produced by applying a Ni plating layer onto the surface of a Cu substrate, applying a Rh plating layer of 1 μm thickness, and then applying a Au plating layer of the same thickness. The sliding contact member shows good durability and small noise production, and is suitable for slip rings used in VTR, radar or measuring devices. It has a smooth surface which decreases wear.

Photomagnetic Recording Medium with Platinum- or Palladium-Cobalt Lattice Film
OKI ELECTRIC IND. CO. LTD.
Japanese Appl. 4/255,934
Two layer photomagnetic recording medium comprises a recording layer and an auxiliary film layer 5–70 mm thick made of a Pt/Co or Pd/Co artificial lattice film. The medium is overwritable, is thin and has improved sensitivity.

Photomagnetic Palladium-Cobalt Alloy Perpendicular Recording Film
SANYO ELECTRIC CO. Japanese Appl. 4/258,828
Photomagnetic Pd-Co alloy recording film of thickness ≤ 300 Å is prepared by sputtering, with the pressure of the vacuum sputtering vessel being at least 5 m Torr. The medium has almost 100% rectangular ratio and H/Hc. In an example, a photomagnetic disk was prepared by sputtering a 300 Å thick alloy film, having Pd-Co ratio 6:4 at.% on a glass substrate.

Silver-Palladium-Platinum Fine Powder Paste
TANAKA KIKINZOKU KOGYO K.K.
Japanese Appl. 4/289,608
The paste comprises 10–20 wt.% Pd, 0.1–4.7 wt.% Pt and balance Ag, which is mixed and made into paste. Resin, organic solvent, glass frit and additive were added, in an example, to the powder mixture to prepare a paste. The paste was screen printed on an Al2O3 substrate, then sintered at 900°C for 30 min, to form a Ag-Pd-Ag alloy thick film circuit. The paste is used in formation of conductive thick film for a circuit without any erosion by soldering.

Electric Switch Contact Element Containing Palladium Alloy
SIEMENS A.G.
German Appl. 4,013,627
A contact element for an electric switch contact consists of a non-porous metal support containing a 2–6 μm thick electroplated or cathodically sputtered Pd-containing contact deposit, with at least the outer layer being formed by ≥ 0.1 μm thick Pd-Ag or Pd-Au alloy layer. The contact element which is used in an electromagnetic relay, is reliable and durable.

MEDICAL USES

Anaerobic Microbiological Culture Vessel Containing Palladium Catalyst
AIR LIQUIDE L.
European Appl. 460,979A
Cultures placed in an anaerobic microbiological culture vessel, which is connected to a gas cylinder and an exhaust pipe, is purged with the CO2, H2 and N2 before incubation. The vessel also contains Pd catalyst which aids the reaction of traces of O2 with H2O to form H2O. The vessel is used for microbiological culture in medical analysis, or in the food industries.

New Arylidene Glycolate Platinum Complex
CHISSO CORP. Japanese Appl. 3/246,251
Arylidene glycolate Pt complexes which optionally contain substituted phenyl, H, and 1–6C alkyl, are prepared by treatment with anion-exchange resin such as “Amberlite IR A400”, and a further reaction with α-hydroxycinnamic acid in H2O, evaporating and washing the residue with an organic solvent. The complex is useful as an anti-cancer agent.

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